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IS 11964 (1987): Method for determination of free acidity in caseins (Reference method) [FAD 19: Dairy Products and Equipment]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
केसीन — मुक्त अम्लता ज्ञात करना
(संदर्भ पद्धति)
(पहला पुनरीक्षण)

Indian Standard
CASEINS — DETERMINATION OF FREE ACIDITY
(REFERENCE METHOD)
(*First Revision*)

ICS 67.100.10

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NATIONAL FOREWORD

This Indian Standard (First Revision) which is identical with ISO 5547 : 2008 'Caseins — Determination of free acidity (Reference method)' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Dairy Products and Equipment Sectional Committee and approval of the Food and Agriculture Division Council.

This standard was first published in 1987 under dual numbering system which was identical with ISO 5547 : 1978. This first revision has been brought about to align it with the latest version of ISO 5547 : 2008 which has been technically revised.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their respective places are listed below along with their degree of equivalence for the editions indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 385 : 2005 Laboratory glassware — Burettes	IS 1997 : 2008 Laboratory glassware — Burettes	Identical
ISO 648 Laboratory glassware — Single volume pipettes	IS 1117 : 1975 Specification for one-mark pipettes	Technically Equivalent
ISO 835 : 2007 Laboratory glassware — Graduated pipettes	IS/ISO 835 : 2007 Laboratory glassware — Graduated pipettes	Identical
ISO 3310-1 Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth	IS 460 (Part 1) : 1985 Specification for test sieves: Part I Wire cloth test sieves	Technically Equivalent
ISO 4788 : 2005 Laboratory glassware — Graduated measuring cylinders	IS 878 : 2008 Laboratory glassware — Graduated measuring cylinders	Identical
ISO 5550 : 2006 Caseins and caseinates — Determination of moisture content (Reference method)	IS 11920 : 2012 Caseins and caseinates — Determination of moisture content (Reference method) (<i>first revision</i>)	do

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard
**CASEINS — DETERMINATION OF FREE ACIDITY
(REFERENCE METHOD)**
(First Revision)

1 Scope

This International Standard specifies a reference method for the determination of the free acidity of caseins obtained by acid precipitation or lactic fermentation and of rennet caseins.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single volume pipettes*

ISO 835, *Laboratory glassware — Graduated pipettes*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ISO 5550 | IDF 78, *Caseins and caseinates — Determination of moisture content (Reference method)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

free acidity of caseins

volume, in millilitres, of a 0,1 mol/l standard volumetric sodium hydroxide solution required to titrate an aqueous extract of 1 g of the product

4 Principle

A test portion at 60 °C is extracted using water and filtered. The filtrate is then titrated against a standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

5 Reagents

Use only reagents of recognized analytical grade, and only distilled or demineralized water or water of equivalent purity, freed from carbon dioxide by boiling for 10 min before use.

5.1 Sodium hydroxide (NaOH), approximately 0,1 mol/l standard volumetric solution.

5.2 Phenolphthalein (C₂₀H₁₄O₄), 10 g/l ethanolic solution.

6 Apparatus

Usual laboratory apparatus, and in particular the following.

6.1 Analytical balance, capable of weighing to the nearest 0,01 g.

6.2 Conical flask, of capacity 500 ml, with ground neck and fitted with a ground-glass stopper.

6.3 One-mark pipette, of capacity 100 ml, complying with the requirements of ISO 648, class A.

6.4 Pipette, suitable for measuring 0,5 ml of indicator solution (5.2), complying with the requirements of ISO 648, class A or ISO 835, class A.

6.5 Conical flask, of capacity 250 ml.

6.6 Measuring cylinder, of capacity 250 ml, complying with the requirements of ISO 4788, class A.

6.7 Burette, graduated in at least 0,1 ml divisions, complying with the requirements of ISO 385, class A.

6.8 Water bath, capable of being maintained at a temperature of $(60 \pm 2) ^\circ\text{C}$.

6.9 Appropriate filter.

6.10 Grinding device, for grinding the laboratory sample, if necessary (see 8.1.4), without development of undue heat and without loss or absorption of moisture. A hammer-mill shall not be used.

6.11 Test sieve, wire cloth, of diameter 200 mm, with nominal size of aperture 500 μm , with receiver, complying with ISO 3310-1.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707|IDF 50 [1].

8 Procedure

8.1 Preparation of the test sample

8.1.1 Thoroughly mix the laboratory sample by repeatedly shaking and inverting the container (if necessary, after having transferred all of the laboratory sample to an airtight container of sufficient capacity to allow this operation to be carried out).

8.1.2 Transfer about 50 g of the thoroughly mixed laboratory sample to the test sieve (6.11).

8.1.3 If the 50 g portion directly passes or almost completely passes the sieve, use for the determination the sample as prepared in 8.1.1.

8.1.4 Otherwise, grind the 50 g portion, using the grinding device (6.10), until it passes the sieve. Immediately transfer all of the sieved sample to an airtight container of sufficient capacity and mix thoroughly by repeatedly shaking and inverting. During these operations, take precautions to avoid any change in the water content of the product.

8.1.5 After the test sample has been prepared, the determination (8.3) should proceed as soon as possible.

Clean the device after grinding each sample.

8.2 Test portion

Weigh about 10 g of the test sample (8.1) to the nearest 10 mg and transfer it to the conical flask (6.2).

8.3 Determination

Using the 250 ml measuring cylinder (6.6), add 200 ml of freshly boiled water, previously heated to 60 °C. Stopper the flask, mix by swirling and place in the water bath at 60 °C (6.8) for 30 min. Shake the flask at intervals of about 10 min.

Filter, and cool the filtrate to about 20 °C. The filtrate must be clear.

Transfer 100 ml of the cooled filtrate into the conical flask (6.5), using the pipette (6.3). Add 0,5 ml of phenolphthalein solution (5.2), using the pipette (6.4). Titrate with sodium hydroxide solution (5.1), until the appearance of a faint pink colour, persisting for at least 30 s. Record the volume used to the nearest 0,01 ml.

9 Expression of results

9.1 Calculation

9.1.1 The free acidity of the casein, V_{fac} , in millilitres, is given by Equation (1)

$$V_{\text{fac}} = \frac{20 V_{\text{NaOH}} c}{m} \quad (1)$$

where

V_{NaOH} is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (5.1) used;

c is the amount of substance concentration, in moles per litre, of the standard volumetric sodium hydroxide solution (5.1);

m is the mass, in grams, of the test portion.

Calculate the free acidity to the nearest 0,01 ml and report the final result to the nearest 0,1 ml.

9.1.2 To calculate the free acidity of the sample on the dry basis, multiply the result obtained from Equation (1) by Factor (2)

$$\frac{100}{100 - w_w} \quad (2)$$

where w_w is the water content, as a percentage by mass, of the sample determined according to ISO 5550 | IDF 78.

9.2 Precision

9.2.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time will in not more than 5 % of cases be greater than 0,02 ml of 0,1 mol/l sodium hydroxide solution per 1 g of product.

9.2.2 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 0,04 ml of 0,1 mol/l sodium hydroxide solution per 1 g of product.

10 Test report

The test report shall specify:

- a) all the information required for the complete identification of the sample;
- b) the sampling method used, if known;
- c) the test method used, together with a reference to this International Standard;
- d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the result(s);
- e) the test result(s) obtained, and if the repeatability has been checked, the final quoted results obtained.

Bibliography

- [1] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: FAD 19 (1964).

Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

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